526 Rec'd PCT/PTO 16 JAN 2001

FORM PTO 1390
(REV. 5-93)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY DOCKET NUMBER
825-00149

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)
09/743833

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP98/04467	17 July 1998	

TITLE OF INVENTION
METHOD FOR THE DECOMPOSITION OF COMPOSITE MATERIALS THAT CONTAIN SYNTHETIC RESINS

APPLICANT(S) FOR DO/EO/US
Dr. Detlef Frank

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☒ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☐ have not been made and will not be made.
8. ☒ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
    ☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:
    Supplemental Transmittal Letter

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U.S. APPLICATION NO. (if known, see 37 CFR 1.5) 09/743833	INTERNATIONAL APPLICATION NO. PCT/EP98/04467	ATTORNEY'S DOCKET NUMBER 825-00149

17. The following fees are submitted:

Basic National Fee (37 CFR 1.492(a)(1)-(5)):

Search Report has been prepared by the EPO or JPO..................	$ 840.00	$ 840.00	
International preliminary examination fee paid to USPTO (37 CFR 1.482)...	$ 670.00		
No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)).........	$ 760.00		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO........................	$ 970.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4)............................	$ 96.00		
ENTER APPROPRIATE BASIC FEE AMOUNT =			

Claims	Number Filed	Number Extra	Rate		
Surcharge of $130.00 for furnishing the National fee or oath or declaration later than ☐ 20 30 Months from the earliest claimed priority date (37 C.F.R. 1.491(3)).				+	
Total Claims	20 - 20 =	0	x $ 18.00		
Independent Claims	2 - 3 =	0	x $ 78.00		
Multiple dependent claim(s) (if applicable)			+ $260.00		
TOTAL OF ABOVE CALCULATIONS =				$ 840.00	
Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity statement must also be filed. (Note 37 C.F.R. 1.9, 1.27, 1.28).					
SUBTOTAL =				$ 840.00	
Processing fee of $130.00 for furnishing the English Translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 C.F.R. 1.492(f)).					
TOTAL NATIONAL FEE =				$ 840.00	
Fee for recording the enclosed assignment (37 C.F.R. 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 C.F.R. 3.28, 3.31). **$40.00** per property +					
TOTAL FEES ENCLOSED =				$ 840.00	
				Amount to be	
				Charged:	

a. ☒ A check enclosed in the amount of $840.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. 01.2000 in the amount of $____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 01.2000. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

ANDRUS, SCEALES, STARKE & SAWALL, LLP
100 East Wisconsin Avenue, Suite 1100
Milwaukee, Wisconsin 53202

E. R. Sawall 01/16/01
Signature Date

Eugene R. Sawall 17,431
Name Reg. No.

09/743833

PATENT

JC07 Rec'd PCT/PTO 16 JAN 2001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of	)	Group Art Unit:
	)	
DR. DETLEF FRANK	)	Examiner:
	)	
Int'l. Appln. No. PCT/EP98/04467	)	METHOD FOR THE DECOMPOSITION OF
	)	COMPOSITE MATERIALS THAT CONTAIN
Int'l. Filing Date: 17 July 1998	)	SYNTHETIC RESINS

PRELIMINARY AMENDMENT

Box PCT Application
Asst. Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to calculation of the filing fee in the above identified application, please amend the application as follows:

IN THE CLAIMS:

Cancel claims 1-10 and add new claims 11-30.

--11. A process for the material decomposition of composites containing artificial resins, which comprises breaking down the molecular structure of the artificial resin component by a chemical reaction with an alkali metal hydroxide at a temperature in the range from 250 to 370°C.

12. A process as claimed in claim 11, wherein the reaction is carried out at a temperature in the range from 260 to 370°C.

13. A process as claimed in claim 12, wherein the reaction is carried out at a temperature in the range from 300 to 350°C.

14. A process as claimed in claim 11, wherein the alkali metal hydroxide is NaOH, KOH or a mixture of NaOH and KOH.

15. A process as claimed in claim 14, wherein a mixture of NaOH and KOH is used, in which the proportion of potassium hydroxide ranges from 3 to 60% by weight.

16. A process as claimed in claim 11, wherein additionally an auxiliary agent is used in the reaction, which improves the wettability of the composite by the alkali metal hydroxide or lowers

the melting point of the alkali metal hydroxide or, under the reaction conditions, acts as a solvent for the artificial resin or its decomposition products.

17. A process as claimed in claim 11, wherein the artificial resin is a polymer which contains in the main chain functional groups which are chemically cleavable.

18. A process as claimed in claim 17, wherein the artificial resin is selected from the group consisting of polyesters, polyamides, polyethers, polyurethanes, polyimides, polyaramides and polycyanate esters.

19. A process as claimed in claim 17, wherein the artificial resin is an epoxy resin.

20. A process as claimed in claim 11, wherein the composite contains a metallic component.

21. A process as claimed in claim 11, wherein the starting material is a composite conventional in the electrotechnique.

22. A process as claimed in claim 21, wherein the starting material comprises electrical components or circuit boards or waste material obtained in the production of electrical components or circuit boards.

23. A process as claimed in claim 22, wherein the electrical components and the circuit boards attached thereto are separated from each other before the reaction.

24. A process as claimed in claim 21, wherein the starting material comprises or consists of a dust produced in the diminution of the starting material.

25. A process as claimed in claim 21, wherein the dust contains fire retarding agents, metals or both.

26. A process as claimed in claim 11, which is carried out in a discontinuous manner in a stirring vessel or a stirring vessel cascade.

27. A process as claimed in claim 11, which is carried out in a continuous manner in an extruder or an extruder cascade or in a twin-screw extruder.

28. A process as claimed in claim 11, wherein the alkali metal hydroxide is used in an amount of at least 50% by weight referred to the weight of the artificial resin

29. A process for the material decomposition of composites comprising electrical components, circuit boards or waste material obtained in the production of electrical components or circuit boards or mixtures thereof, containing polyimides, polyaramides, polyisocyanate esters, epoxy resins or mixtures thereof which comprises breaking down the high molecular structure of

said resins by a chemical reaction with NaOH, KOH or a mixture thereof at a temperature in the range from 250 to 350°C.

30. A modification of the process of claim 11, wherein the components produced by the reaction are separated from each other and recovered for further use.--

Respectfully submitted,

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09/743833
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METHOD FOR THE DECOMPOSITION OF COMPOSITE MATERIALS THAT CONTAIN SYNTHETIC RESIN

DESCRIPTION

INTRODUCTION

In Germany about 1.5 million metric tons of electronic components are discarded annually, and printed circuit boards account for about 200,000 tons of this scrap material. Because of their complex structure and the toxic substances they contain, such circuit boards are among the most problematic components of electronic scrap with regard to processing for further utilization, and the valuable substances they contain are a resource that has hardly been exploited as yet. The disposal methods in current use, such as dumping or incineration, are extremely problematic because they either introduce toxic heavy metals such as copper, tin or lead into the leakage water or, when bromine-containing flame-proofing agents are incinerated, can produce both aggressive acids and aromatic dioxins and furanes. For recovery of the metals from circuit-board scrap by the methods previously in use, it is often necessary to use elaborate procedures that present difficulties regarding protection of the environment.

STATE OF THE ART

A circuit board ordinarily bears a large assembly of components, the most problematic of which (e.g., batteries, capacitors, rectifiers and mercury switches) are removed before processing. Particularly valuable elements such as gilded connector strips are also removed in advance. This partial disassembly is absolutely necessary for an environmentally tolerable reutilization or disposal, because

all the methods employed so far presuppose that at least a rough breakdown will be undertaken in advance. Otherwise the harmful and valuable materials would become mingled, which would both make recovery more difficult and also possibly entail the release of toxins. The risk of emission of harmful agents could be reduced only by carrying out all the processing steps in hermetically sealed apparatus, which would naturally be expensive.

The proportions by weight of the components and the circuit board from which they have been removed are about equal (45% : 55%). A preparatory separation of all components from the circuit board is reasonable because the components contain about 95% of the chromium and 85% of the nickel and iron, whereas in the circuit board about 80% of the tin, copper and lead is found. As a rule such a complete disassembly is not yet undertaken in practice, although the necessary procedures are known (DE-PS 42 05 405, DE-OS 41 31 620). Therefore circuit boards completely free of components are usually manufacturer's rejects.

A circuit board with components removed consists mainly of metal (30% by weight), glass fiber (50%) and polymer resin (20%), of which only the metals have so far been regarded as valuable. In order to recover these, they must first be concentrated and separated as well as possible from the residual materials. For this purpose several methods exist, which can also be combined with one another.

According to one procedure the circuit board is coarsely fragmented by mechanical means (shredded) to facilitate handling and is then freed from ferromagnetic parts by magnetic separators. Subsequently a fine comminution is carried out, by any of several means:

- In the standard crushing method thermal stress can cause the formation of polybrominated aromatic dibenzodioxins

(PBDD) and polybrominated dibenzofurans (PBDF) from the flame-proofing agents.

- In the cryo-crushing method the crushing is carried out at temperatures so low that the material becomes brittle, which prevents the formation of thermal decomposition products. These advantages are counteracted by higher energy costs, however, for which it is irrelevant whether the cooling is direct (e.g., with liquid nitrogen) or indirect by way of a refrigeration cascade.

- By partial cooling, the region between the normal and cryo methods is covered.

- In the ultrasound method the composite material is broken up by ultrasound, which produces particle-size distributions specific to the materials. This method is very expensive but allows up to four different metal fractions to be isolated.

After comminution the components are separated according to density, grain size or magnetic or electrical properties. For this purpose sifting and sorting apparatus is used as well as magnetic, eddy-current and electrostatic separators. Losses of valuable materials cannot be prevented, because the fine metal particles are distributed over all the fractions. A special problem is presented by the considerable amounts of heavy-metal-containing dust that are produced, because some of it gets into the lungs and can damage health.

- In another procedure, the wet-crushing process, the circuit boards are crushed while damp, which both protects them from thermal stress and also avoids the production of dust. The subsequent separation of valuable materials is usually done by a method similar to flotation, followed by drying.

The metal-rich fraction is further processed by chemical or thermal means.

- If the content of precious metals is greater than 0.02%, it is worthwhile to prepare the material for precious-metal separating plants. In this process the precious metals are either leached out with a weakly alkaline cyanide solution and then recovered by reduction, or extracted thermometallurgically at 1000-1200°C in a shaft furnace with lead. Subsequently the lead is removed again as litharge in a refining furnace. In both cases the processing is electrolytic and the residues, after the precious metals have been removed, are passed on to copper works.

- If the content of precious metals is less than 0.02% by weight, the material is transferred to the second stage of the copper-production process. Here the raw copper is freed of iron in a drum-type furnace (Pierce Smith converter) with the addition of silicate, so as to produce an iron silicate slag that permanently encloses other heavy metals and can be used as a building material (cobblestone pavement). The copper is then cleaned electrolytically, leaving the precious metals in the anode slime.

Attempts at chemical recovery by solvolysis of the polymer have so far been defeated by the long reaction times and the high costs of the process, but it is possible to use this approach both with concentrated nitric acid at room temperature and with suitable solvents at higher temperatures in an autoclave.

All the physical procedures just described exhibit the same general conflict of objectives. On one hand the material must be broken down into very fine fragments so that the individual components can be separated from one another; but the finer the powder becomes, the more difficult it is to

achieve separation because surface effects level out the differences between the materials. On the other hand, there is necessarily a trade-off between the purity of a fraction and the yield. Another problem resides in the fact that the metal-poor fraction must be disposed of together with the dust fraction and accounts for more than 2/3 of the total amount. Ordinarily this waste is deposited in an ordinary dump, although the powdery nature of the material and the residual heavy-metal contamination actually should make it obligatory to dispose of it in a dump or incinerator designed for special kinds of waste.

In all the separation procedures mentioned above, contamination of the metal fraction with polymers and glass is a problem, making it necessary to decompose the material before the metal can be electrolytically purified. Furthermore, the metal content of the residual fraction prevents it from being reclaimed for further use and also makes disposal more difficult.

The patent US-PS 5 580 905 describes a method for the hydrolytic splitting of polyesters, specifically polyalkylene terephthalates, by means of caustic solutions of alkali hydroxides, in which the mixture is heated and the polyester is decomposed to produce the associated salt plus polyhydroxy alcohol. The reaction occurs basically at temperatures up to the boiling point of the alcohol, i.e. up to ca. 200°C, in order to separate the alcohol by vaporizing it. The use of powdery or molten hydroxides described in column 6/30-32 should be considered in connection with the disclosure of the cited US-PS as a whole, namely that by this means the required solutions of alkali hydroxides are formed directly in the reaction mixture and that the operating temperatures are such that the alcohol can be vaporized and distilled off. The subsequent heating to distinctly higher temperatures likewise described, in column 4/7-11, clearly no longer refers to the decomposition of the polyester material but rather to the thermal breakdown of

impurities under conditions such that the alkali terephthalate is still stable.

Furthermore, the patent DE-OS 4 001 897 discloses a method for disintegrating (circuit) boards in which the boards are put into a molten mixture of alkali hydroxide and alkali oxide at temperatures above 400°C, with the exclusion of oxygen. Then when oxygen is added, the alkali oxide is converted to peroxide, which subsequently diffuses into the molten mixture and breaks down oxidizable components derived from the boards, such as carbon- or hydrogen-containing compounds. In this process the mixture of alkali hydroxide and alkali oxide is used as a matrix for the oxidation (column 1/25-32) and prevents the formation of toxic gases such as would be produced during combustion. As disclosed in column 1/29-34, it is not possible to salvage as raw material and reutilize either the synthetic resin or the glass fibers.

INVENTION

The objective of the invention is to develop a method for decomposing the composite material comprising metal, glass and polymer. The isolation of largely pure metal, glass and polymer fractions is intended to enable them to be further processed, with the aim of substantially reducing or to a great extent avoiding the production of residual fractions that would have to be treated as waste.

The subject matter of the invention is a method for separating the components of composite materials containing synthetic resin, in which the structure of the high-molecular-weight synthetic resin is broken down by chemical reactions in molten alkali metal hydroxides at temperatures above 250°C.

The preferred hydroxides of alkali metals are NaOH and KOH and, most especially, mixtures of NaOH and KOH. In such

mixtures the proportion of potassium hydroxide, for example, ranges from 3 to 60% by weight, preferably 5-20%. The relatively small contribution of potassium hydroxide is determined by, among other things, the higher price of this material.

The reaction temperatures are in general in the range between 260 and 400°C, preferably in the range between 280 and 370°C, and most preferably in the range between 300 and 350°C. The choice of most suitable temperature naturally depends on the nature and composition of the materials to be treated, and it should be kept in mind here that at higher temperatures both the speed of the reaction and the danger of thermal formation of undesirable breakdown products are increased. In general, moreover, within the above-mentioned temperature ranges the use of potassium rather than sodium hydroxide makes it possible to work at a lower temperature, and when mixtures of sodium and potassium hydroxide are used, the temperature can be lowered still further.

The reaction can be enhanced by employing accessory chemicals that facilitate wetting of the composite material by alkali metal hydroxides, lower the melting point of the alkali metal hydroxides, or have solvent or swelling effects on the synthetic resin or its decomposition products under the conditions of the reaction.

Accessory chemicals that can be considered for improving wettability of the material, for example, are surfactants that are stable at the reaction temperatures. Examples of those that reduce the melting point of the alkali metal hydroxides include inorganic salts of alkaline, alkaline earth or earth metals, of metals belonging to the fourth group of the periodic table, or of metals belonging to neighboring groups with strong or weak inorganic acids. Addition of salts of metals that are already present in the materials to be treated can also enhance the reaction. This also offers the advantage that no foreign elements are

introduced to the system. Among the accessory chemicals that may be used to assist solution or swelling of the synthetic resin or its breakdown products are oligomeric fragments or intermediate constituents of the synthetic resin that are stable under the reaction conditions. For example, when materials containing epoxy resins are to be decomposed, the phenolic intermediates bisphenol A and F (4,4' diphenylol-2,2-propane and -methane, respectively) may be considered.

The synthetic resins are cross-linked or un-cross-linked polymers, the main chain of which contains functional groups that can be split chemically, such as polyesters, polyamides, polyethers, polyurethanes, but most commonly used are polyimides such as polyphthalimides and poly-bimalinimides, polyaramides and polycyanate esters, but especially epoxy resins. These as a rule consist of condensation products of bisphenols, such as bisphenol A and bisphenol F, and epichlorhydrin. This list is exemplary and should not be understood as restrictive. Restrictions can be inferred from the nature of the chemical breakdown and will be obvious to those skilled in the art.

The amount of alkali metal hydroxide to be employed can be varied within broad limits. Of course the amount used in practice must be at least sufficient to ensure that the process is completed. For example, although it would suffice to add alkali metal hydroxide in a quantity amounting to 50% by weight of the synthetic resin, a considerably larger amount is used in order to facilitate handling.

Because the method is very simple, it can easily be applied to other composite materials. It is especially advantageous to use it for metallic components in the composites customary in electrical technology, which are present, for example but not exclusively, in circuit boards and their components or rejects produced in the manufacture thereof. The term "component" is used here in particular for all the electronic devices that are or could be mounted on printed

circuit boards, such as processors, memory chips, resistors and capacitors. This list is exemplary and should not be understood as restrictive. In this regard it is reasonable but not necessary for the components mounted on the circuit boards to be separated from the boards before the reaction and, where appropriate, processed separately altogether or in part, which can occur according to the conventional methods. The separation can be done, e.g., by chemical (tin/lead stripping), thermal (unsoldering) or mechanical ("shaving off") procedures.

For the comminution of composite materials such as circuit boards, commercially available shredder equipment can be used. Because the fragments need not be extremely small, this part of the process is not very demanding. The maximal tolerable fragment size will be inferred by the person skilled in the art from the requirements imposed by subsequent stages in the procedure.

During dry comminution of composite materials according to the state of the art, as is generally known, dust-like components are also produced which are separated out by filtering and disposed of. The method in accordance with the invention now offers the advantage that such dusty waste can also be treated to separate its components, either by itself or in combination with the comminuted material. It is a particular advantage of the invention that it is also suitable for the processing of dusts that contain flame-proofing agents and/or metals, because these are frequently present in comminuted materials of the kind customarily used in electrical technology.

For the chemical decomposition of the synthetic resin commercially available reactors can be used. These are mainly agitator boilers or cascades thereof in the case of discontinuous operation, or extruders and extruder cascades, e.g. a twin-screw extruder, for continuous operation. The substantial advantage of the extruder lies in the brief and

well-defined reaction time. The danger of formation of thermal decomposition products (dioxins and carbonized materials) is therefore substantially less than when an agitator boiler is used. On the other hand, long sojourn times are impossible with an extruder, or are possible only with costly extruder cascades. Experiments have indicated the reaction times (examples 1 to 3) that can be achieved with an extruder. Because cleaning of the reaction space is also simpler with an extruder and not even necessary in the case of continuous operation, the extrusion process is preferred, and the twin-screw extruder is particularly suitable because it produces better mixing.

So that the components of the composite materials can be recycled, they must be separated from one another after decomposition is complete. This can occur in the customary way, for example by dry (pneumatic sifting, electrostatic) or wet (flotation) means. In both cases it is possible to use commercially available equipment. The advantage of the dry methods resides chiefly in the lower water and energy consumption, whereas the wet method makes it simpler to avoid the emission of dusts. For separating the breakdown products of the synthetic resin and the reclamation of excess reagent, extraction processes employing organic and aqueous solvents are preferred. Here, again, commercially available equipment can be used. Someone skilled in the art can select suitable methods and apparatus for extraction with no difficulty.

Examples 1 to 3

A bare printed circuit board, i.e. one on which no components were mounted, on epoxy-resin base of the class FR-4, i.e. a flame-resistant product, was coarsely comminuted so that the resulting fragments had an area of 20x20 mm and a thickness of 1.6 mm. To the comminuted laminate was added the same weight of alkali hydroxide, and

the mixture was put into a temperature-controlled molten-metal bath. Then the reaction proceeded under the following conditions: example 1, addition of NaOH, 340°C; example 2, KOH, 320°C; example 3, mixture of NaOH and KOH in equal amounts by weight, 300°C. In all cases the reaction was completed, with the development of gas, in less than 5 minutes. The gas produced was condensed as water. After the reaction had ended, the mixture was cooled and washed with cold water until the washing water was approximately neutral. After separation and removal of the decomposed polymer and subsequent drying, it was a simple matter to separate the metal and glass fractions from one another.

CLAIMS

1. Method for the decomposition of composite materials containing artificial resins, characterized in that the molecular structure of the artificial resin component is broken down by chemical reaction with alkali metal hydroxides at temperatures above 250°C, advantageously at temperatures between 260 and 400°C, preferably in the range between 280 and 370°C, and especially preferably in the range between 300 and 350°C.

2. Method according to Claim 1, characterized in that the alkali metal hydroxide is NaOH or KOH, but preferably a mixture of NaOH and KOH.

3. Method according to Claim 1 or 2, characterized in that the reaction is facilitated by auxiliary agents that improve the wettability of the composite material by the alkali metal hydroxides, lower the melting point of the alkali metal hydroxides, or under the reaction conditions act as a solvent for the artificial resin or its decomposition products.

4. Method according to one or more of the claims 1 to 3, characterized in that the artificial resins are cross-liked or un-cross-linked polymers that contain in their main chain chemically cleavable functional groups such as polyesters, polyamides, polyethers, polyurethanes, preferably polyimides, polyaramides and polycyanate esters and in particular epoxy resins.

5. Method according to one or more of the claims 1 to 4, characterized in that the composite material contains metallic components.

6. Method according to one or more of the claims 1 to 5, characterized in that the starting material consists of a composite conventionally used in electronic technology, in particular circuit boards, components or waste material produced in the manufacture of circuit boards or components, and preferably the circuit boards are separated from the components attached thereto prior to the reaction.

7. Method according to Claim 6, characterized in that the starting material consists of the dusts produced during comminution or contains these dusts, in particular those that comprise flame-retardant agents and/or metals.

8. Method according to one or more of the claims 1 to 7, characterized in that the reaction is carried out discontinuously in an agitator vessel, preferably in an agitator-vessel cascade, or continuously in an extruder or an extruder cascade, preferably in a twin-screw extruder.

9. Method according to one or more of the claims 1 to 8, characterized in that the alkali metal hydroxide is present in an amount equivalent to 50% by weight of the artificial-resin component.

10. A modification of the method according to one or more of the claims 1-9, characterized in that the components of the composite material are separated from one another after the reaction and where appropriate are partially or entirely recovered for further use.

Translation of PCT/EP98/04467

ABSTRACT

The invention relates to a method for the decomposition of composite materials that contain synthetic resin, characterized in that the structure of the high-molecular-weight resin component is broken down by chemical reaction with hydroxides of the alkali metals at temperatures above 250°C, with subsequent reprocessing where appropriate, in whole or in part.

PCT WELTORGANISATION FÜR GEISTIGES EIGENTUM
Internationales Büro

INTERNATIONALE ANMELDUNG VERÖFFENTLICHT NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS (PCT)

(51) Internationale Patentklassifikation 6 : **A62D 3/00**	**A1**	**(11) Internationale Veröffentlichungsnummer:** **WO 00/03764** **(43) Internationales Veröffentlichungsdatum:** 27. Januar 2000 (27.01.00)

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(81) Bestimmungsstaaten: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO Patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), eurasisches Patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), europäisches Patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI Patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Veröffentlicht
Mit internationalem Recherchenbericht.

TITLE V

(54) Title: METHOD FOR THE MATERIAL DECOMPOSITION OF COMPOSITES CONTAINING ARTIFICIAL RESIN

(54) Bezeichnung: VERFAHREN ZUM STOFFLICHEN AUFSCHLUSS VON KUNSTHARZ ENTHALTENDEN VERBUNDWERKSTOFFEN

(57) Abstract

The invention relates to a method for the material decomposition of composites containing artificial resin, characterized in that the high–molecular structure of the artificial resin component is broken down by a chemical reaction with hydroxides of the alkali metals at temperatures above 250 °C and possibly partly or fully reprocessed.

(57) Zusammenfassung

Die Erfindung bezieht sich auf ein Verfahren zum stofflichen Aufschluß von Kunstharz enthaltenden Verbundwerkstoffen, das dadurch gekennzeichnet ist, daß die hochmolekulare Struktur des Kunstharzanteils durch chemische Reaktion mit Hydroxiden der Alkalimetalle bei Temperaturen oberhalb von 250 °C abgebaut wird und gegebenenfalls teilweise oder ganz aufgearbeitet werden.

Type a plus sign (+) inside this box [+]

Approved for use through 9/30/00
Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE

PTO/SB/01 (8/96)

DECLARATION

☐ Declaration Submitted with Initial Filing OR ☒ Declaration Submitted after Initial Filing

Attorney Docket Number	825-00149
First Named Inventor	Detlef Frank
COMPLETE IF KNOWN	
Application Number	09/743,833
Filing Date	01/16/01
Group Art Unit	
Examiner Name	

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD FOR THE DECOMPOSITION FOR COMPOSITE MATERIALS THAT CONTAIN SYNTHETIC RESINS

(Title of the Invention)

the specification of which
☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY) 01/16/01 as United States Application Number or PCT International Number (if applicable). 09/743,833 and was amended on (MM/DD/YYYY) ____

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Copy Attached? YES	NO
			☐	☐	☐
PCT/EP98/04467	PCT	07/17/98	☒	☐	☐
			☐	☐	☐
			☐	☐	☐
			☐	☐	☐

☐ Additional foreign application numbers are listed on a supplemental priority sheet attached hereto:

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	
		☐ Additional provisional Application numbers are listed on a supplemental priority sheet attached hereto.

Type a plus sign (+) inside this box [+]

DECLARATION

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s), or §365© of any PCT international application designated the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States of PCT International application in the manner provided by the first paragraph of Title 35, United States Code §112. I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority sheet attached hereto.

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor: ☐ A petition has been filed for this unsigned inventor

Given Name (first and middle [if any])	Family Name or Surname
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Inventor's Signature: [signature] Date: 10. März 01

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☐ Additional inventors are being named on supplemental sheet(s) attached hereto.